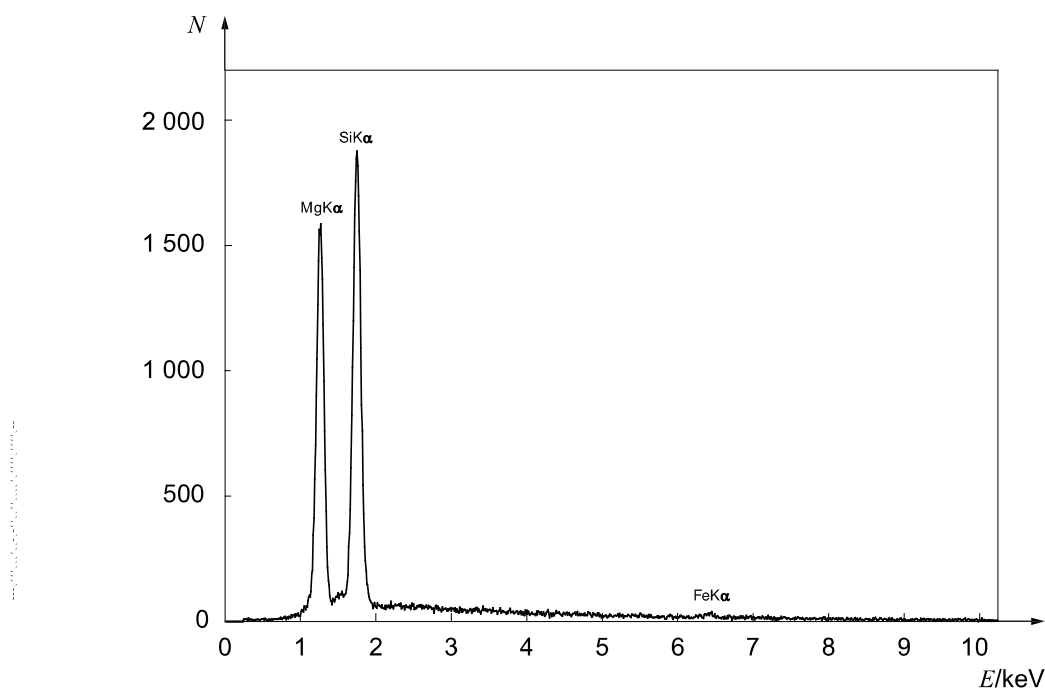


Annex E (normative)

Asbestos identification by SEM in commercial materials

Figures E.1 to E.11 are examples of EDXA spectra collected on an SEM operating at 15 kV and using a silicon solid-state detector with a beryllium window. The SEM specimens were prepared by mounting representative fibre bundles from SRM 1866, SRM 1867, and the HSE reference asbestos varieties on adhesive tape on SEM specimen stubs. All specimens were carbon coated in a vacuum evaporator.

Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



Key

N counts E X-ray energy

Figure E.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile

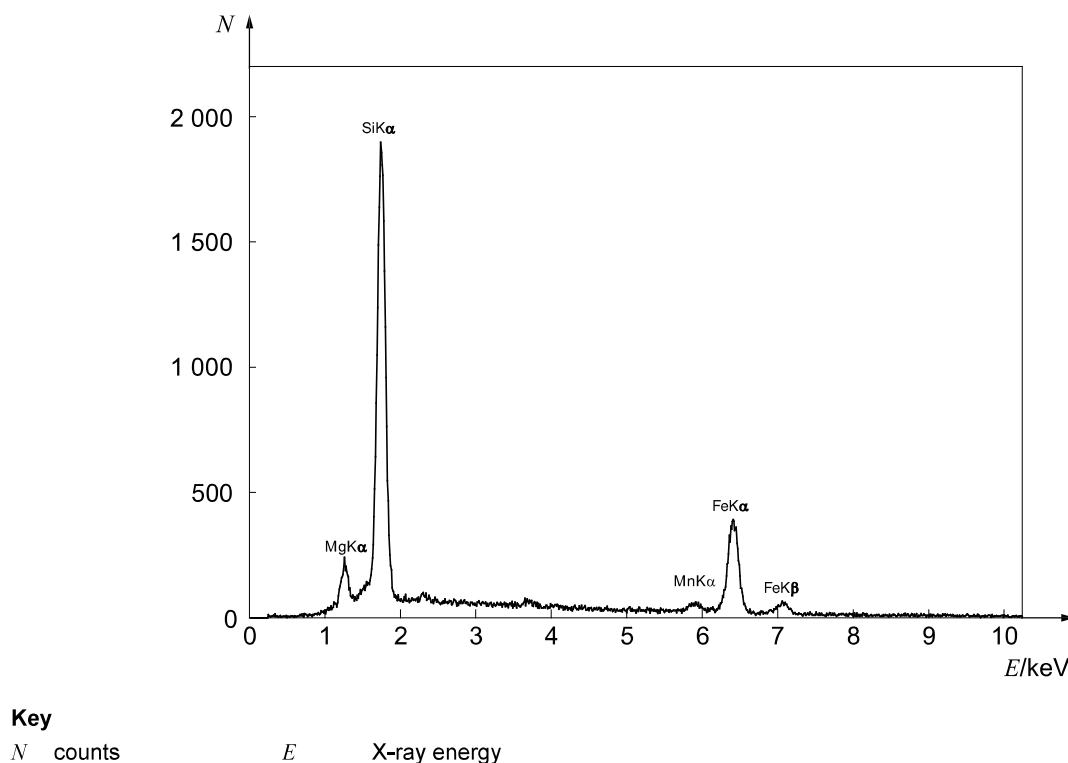


Figure E.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite

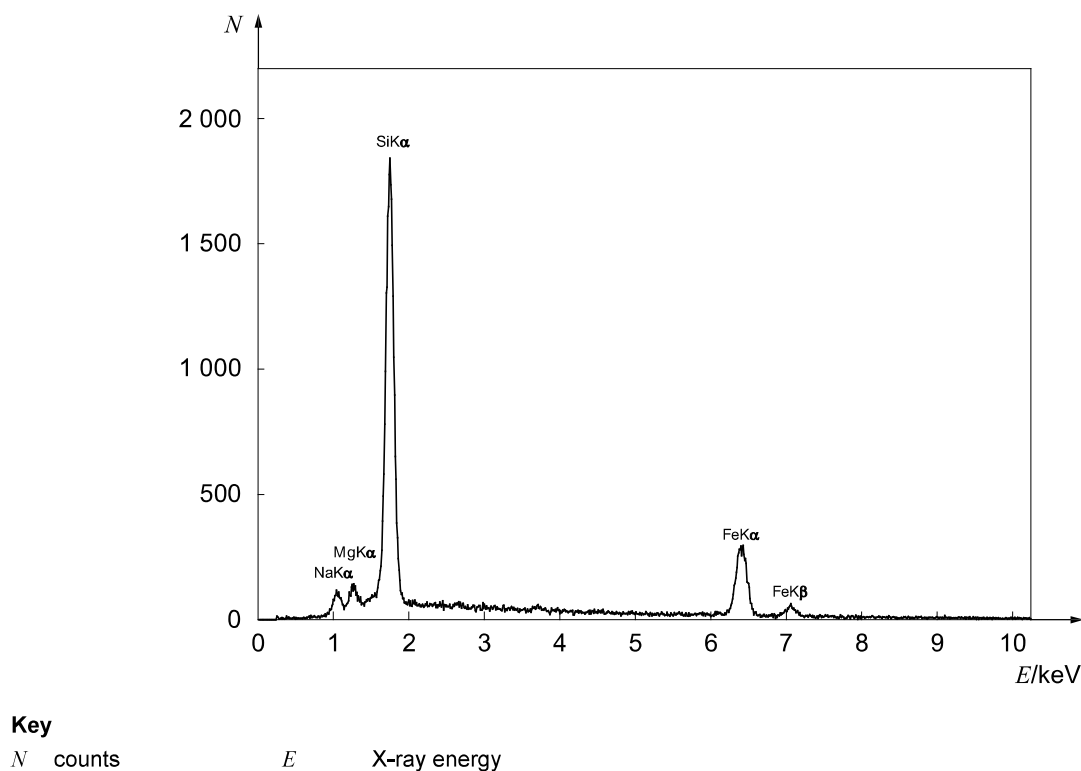
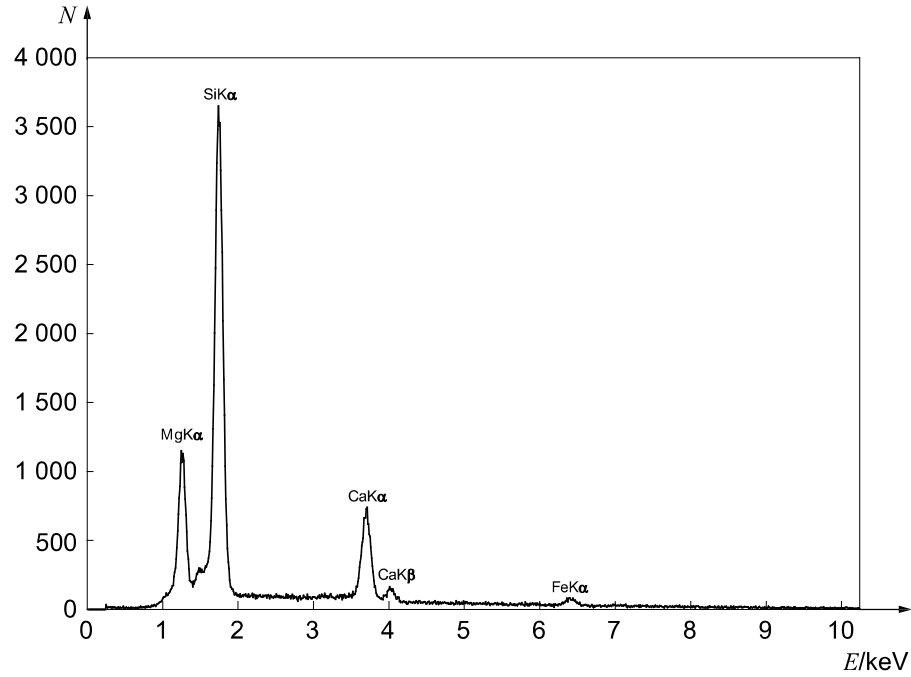


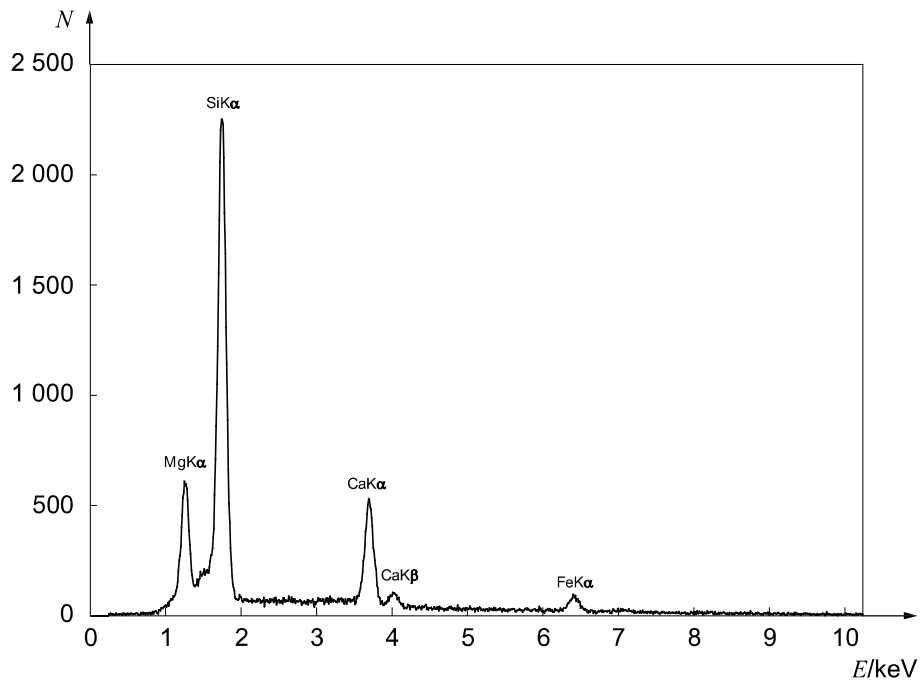
Figure E.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite



Key

N counts E X-ray energy

Figure E.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite



Key

N counts E X-ray energy

Figure E.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite

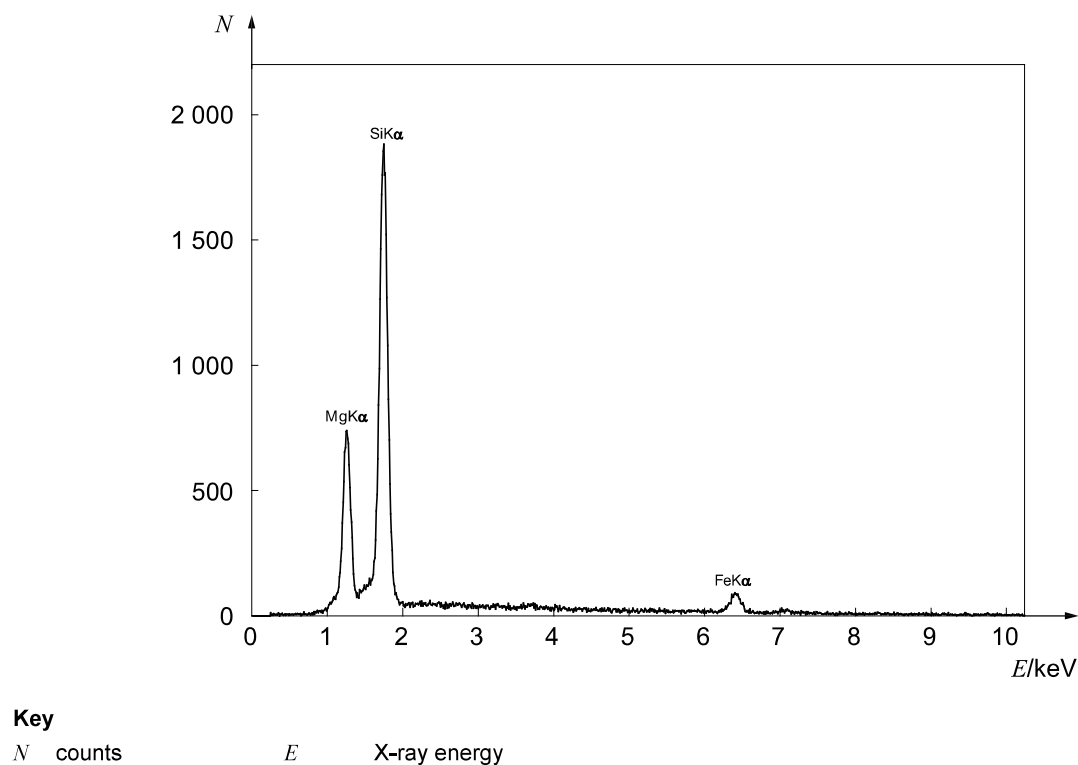


Figure E.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite

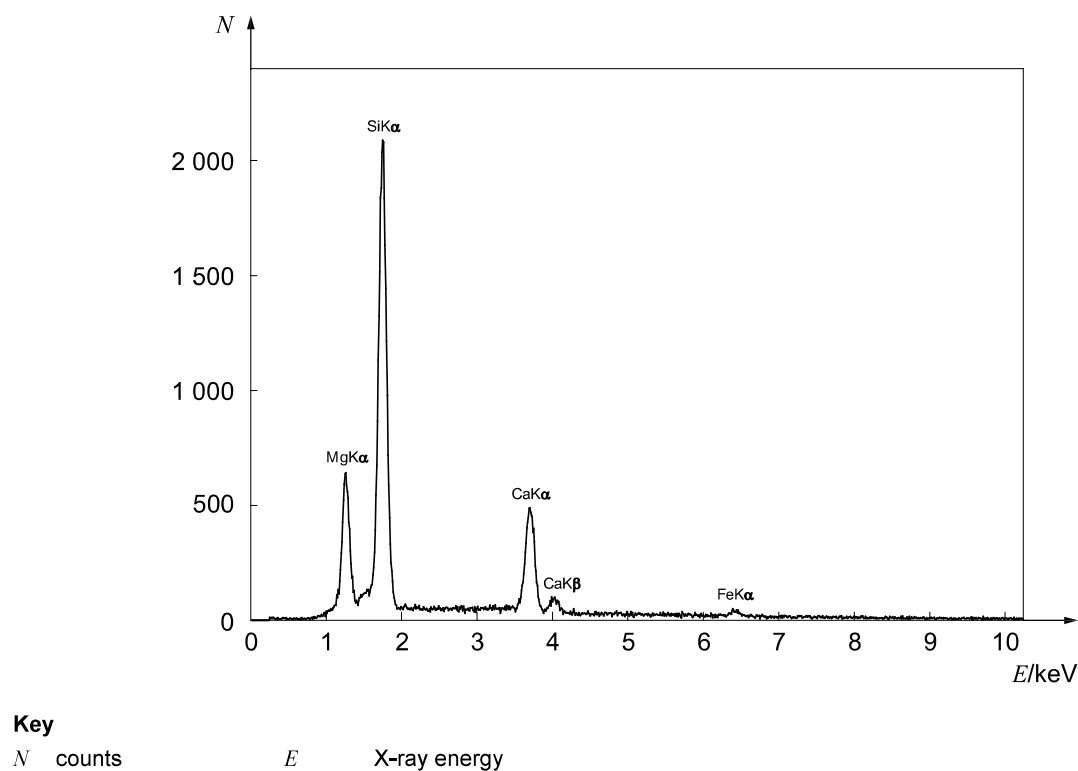
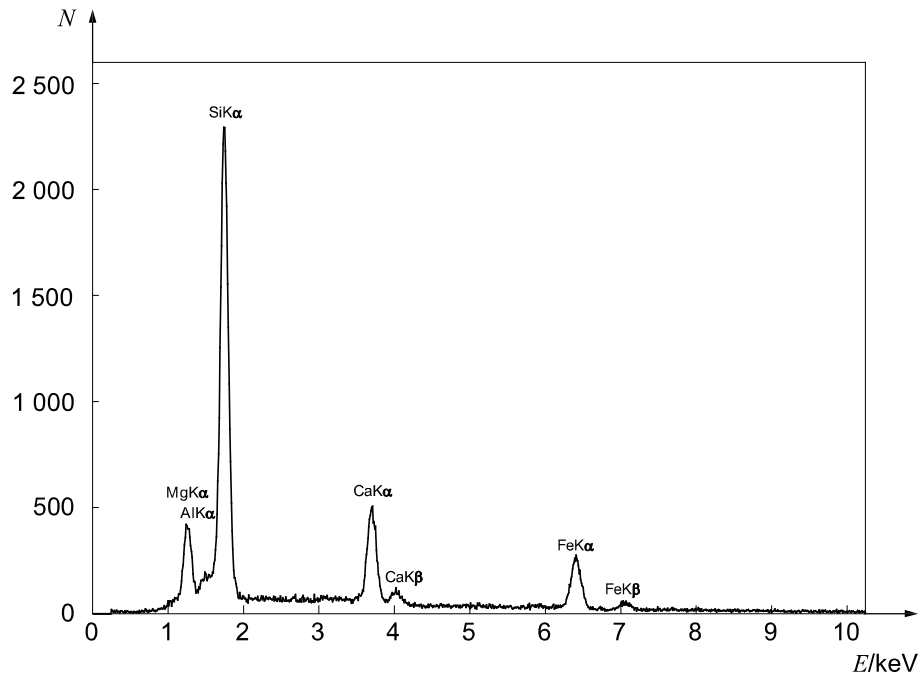
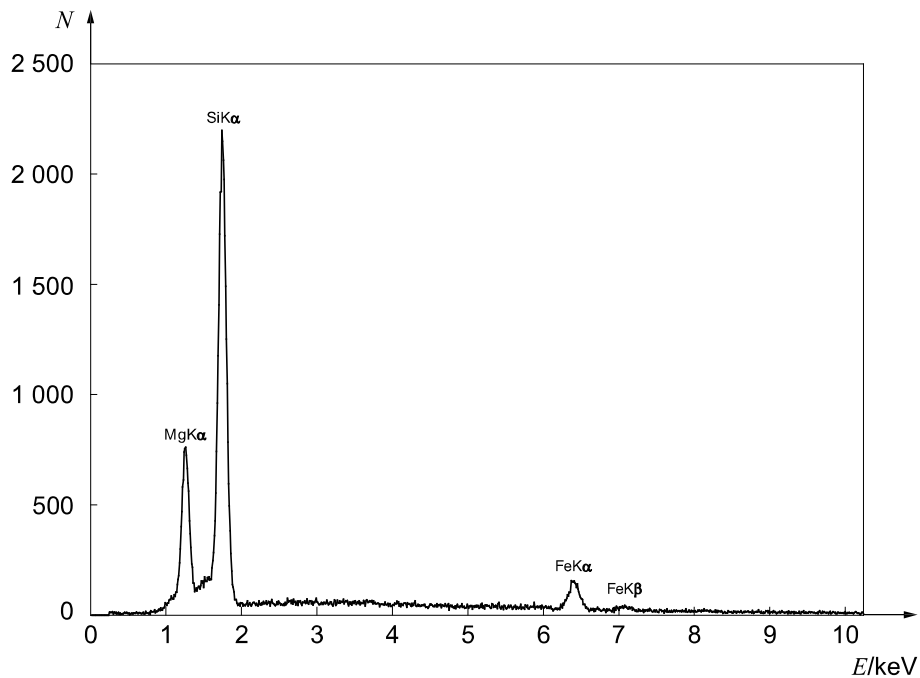


Figure E.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite



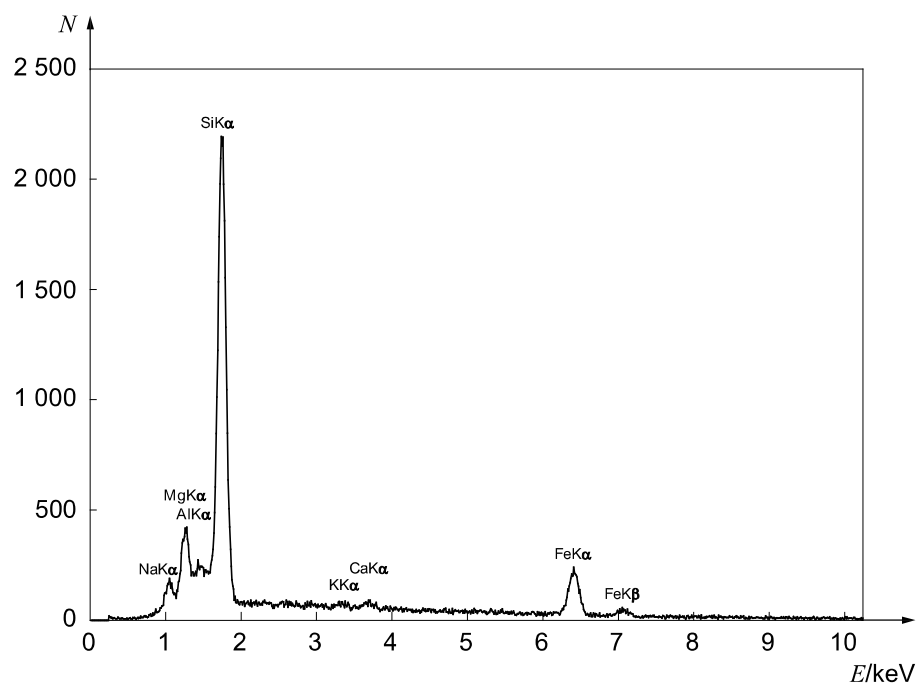
Key
 N counts E X-ray energy

Figure E.8 — Energy dispersive spectrum obtained from HSE actinolite



Key
 N counts E X-ray energy

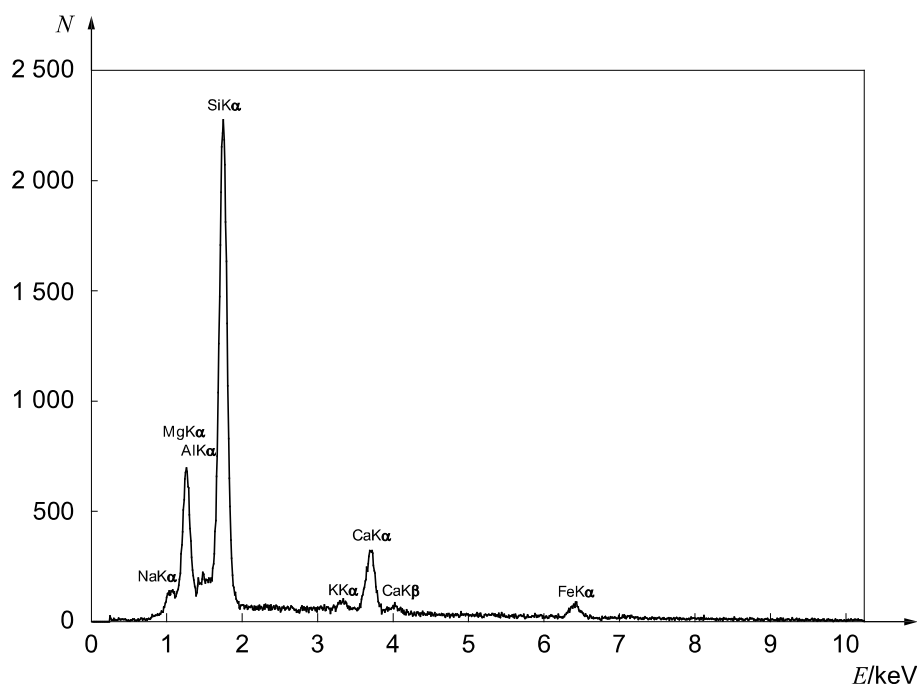
Figure E.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite

**Key**

N counts

E

X-ray energy

Figure E.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite**Key**

N counts

E

X-ray energy

Figure E.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite

Annex F (normative)

Asbestos identification by TEM in commercial materials

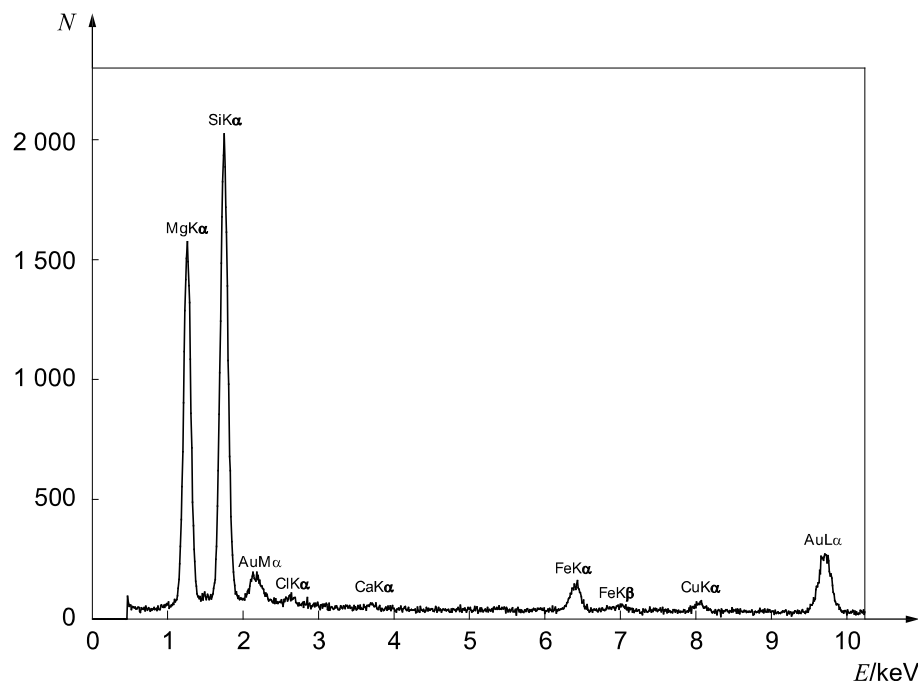
F.1 General

For the identification of asbestos in some types of bulk materials, particularly for those in which PLM examination yields ambiguous results, TEM examination can usually resolve the ambiguities and provide definitive identification of the fibres. In most cases, acquisition of an EDXA spectrum provides sufficient evidence to identify any of the asbestos varieties. Discrimination between talc and anthophyllite, however, cannot be reliably achieved on the basis of an EDXA spectrum alone, because the chemical compositions of the two minerals are very similar. Electron diffraction permits discrimination between talc and anthophyllite on the basis of their different crystal structures.

F.2 EDXA analysis

Figures F.1 to F.11 are examples of EDXA spectra collected on a TEM operating at 80 kV and using a silicon solid state detector with a beryllium window. The TEM specimens were prepared by the micropipette method from SRM 1866, SRM 1867 and HSE reference asbestos varieties. All specimens were prepared using gold grids in order to avoid interference in detection of the Na K_{α} peak by the Cu L_{α} peak which would partially overlap the sodium peak if copper specimen grids were used.

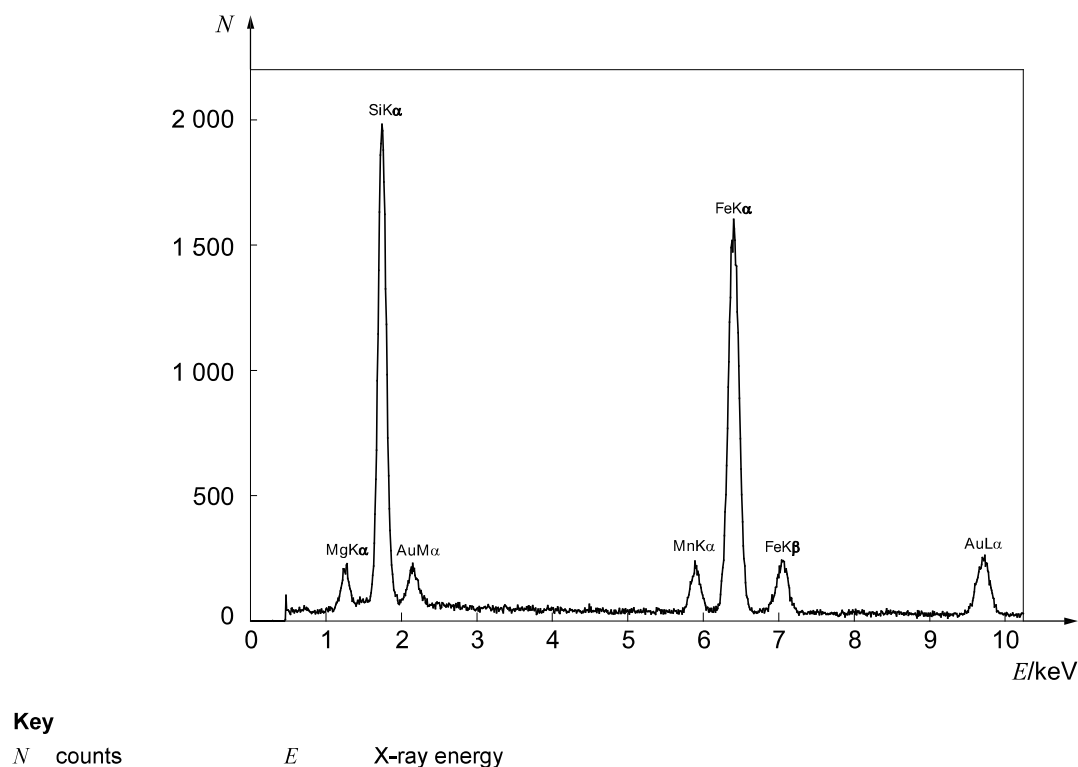
Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



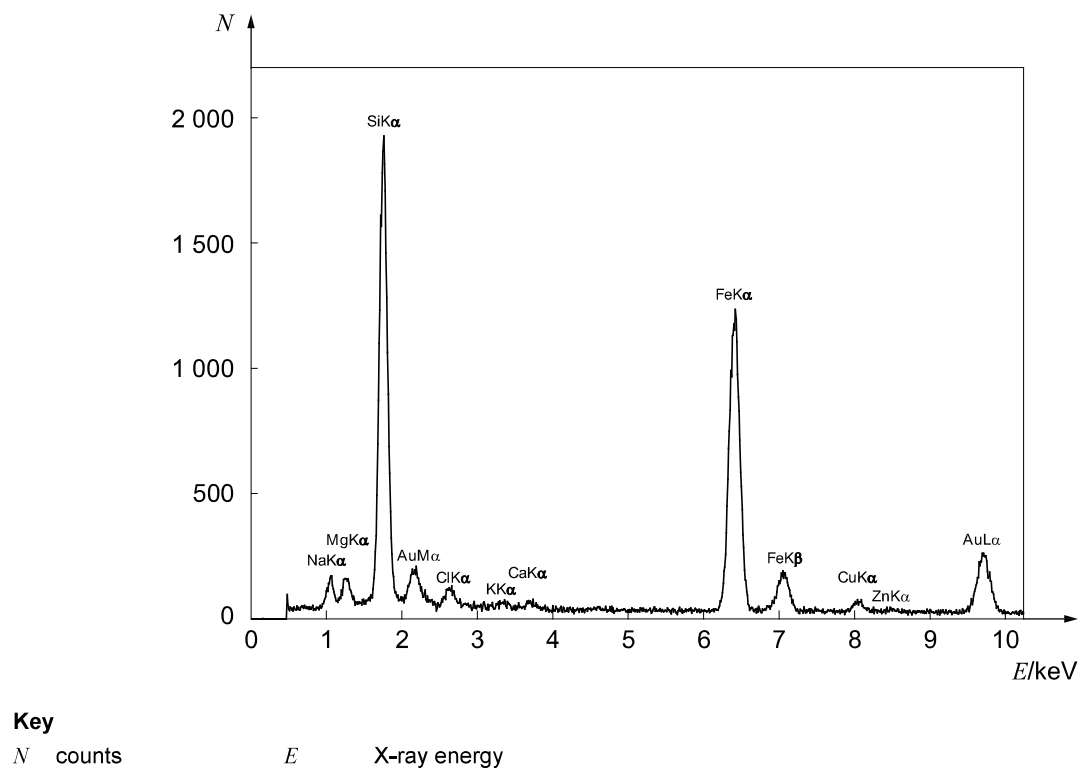
Key

N counts E X-ray energy

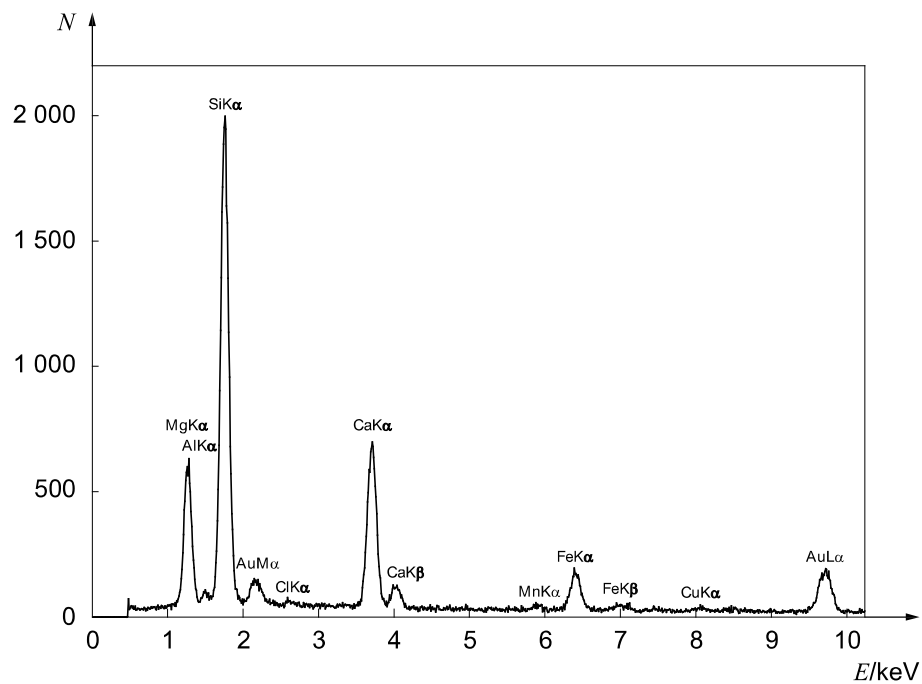
**Figure F.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile.
The gold and small copper peaks originate from the gold specimen grid**



**Figure F.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite.
The gold peaks originate from the gold specimen grid**

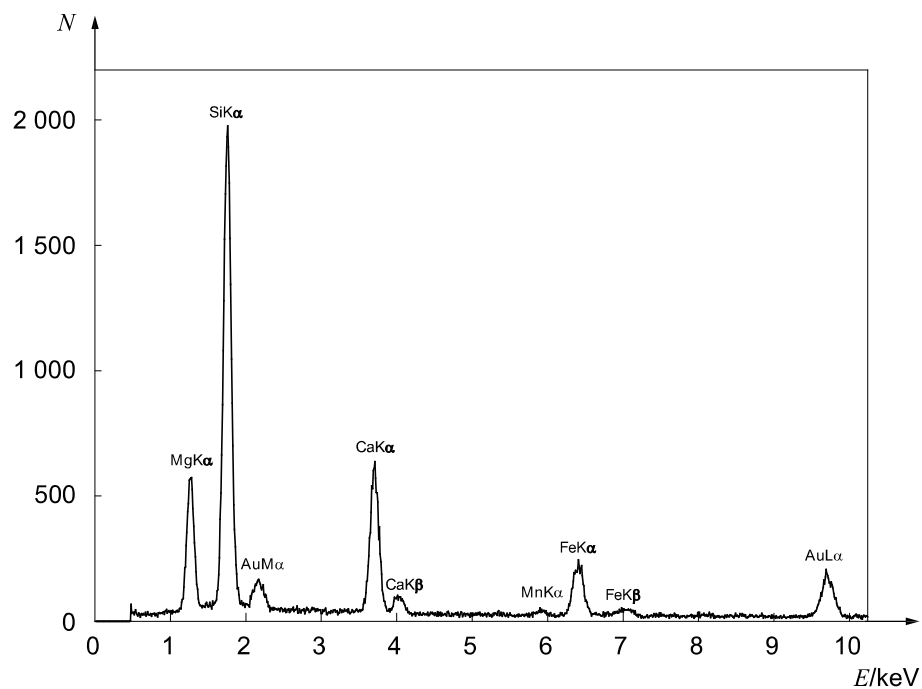


**Figure F.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite.
The gold and small copper peaks originate from the gold specimen grid**



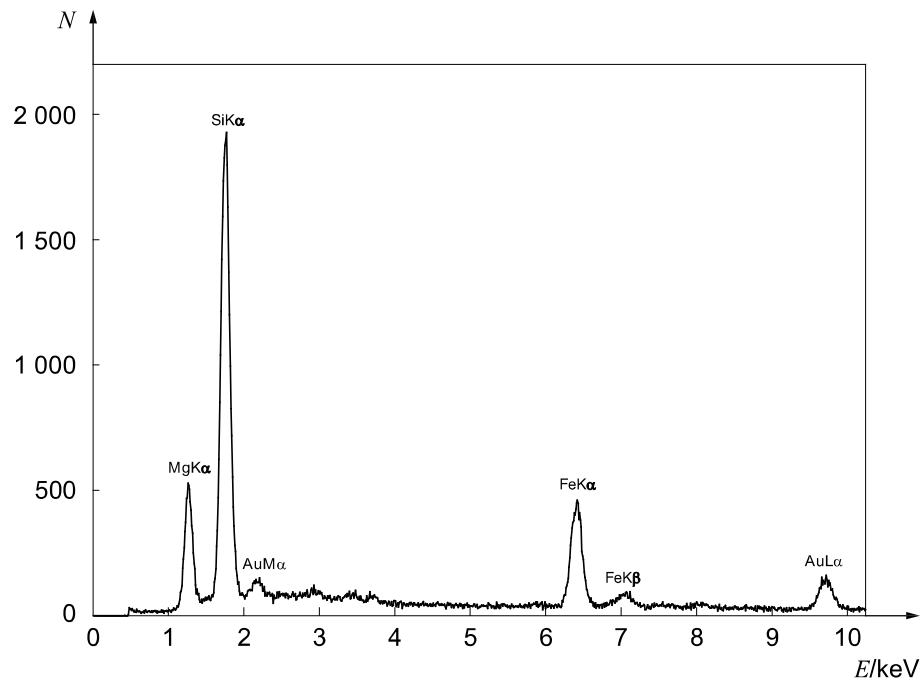
Key
 N counts E X-ray energy

**Figure F.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite.
 The gold and small copper peaks originate from the gold specimen grid**



Key
 N counts E X-ray energy

**Figure F.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite.
 The gold peaks originate from the gold specimen grid**

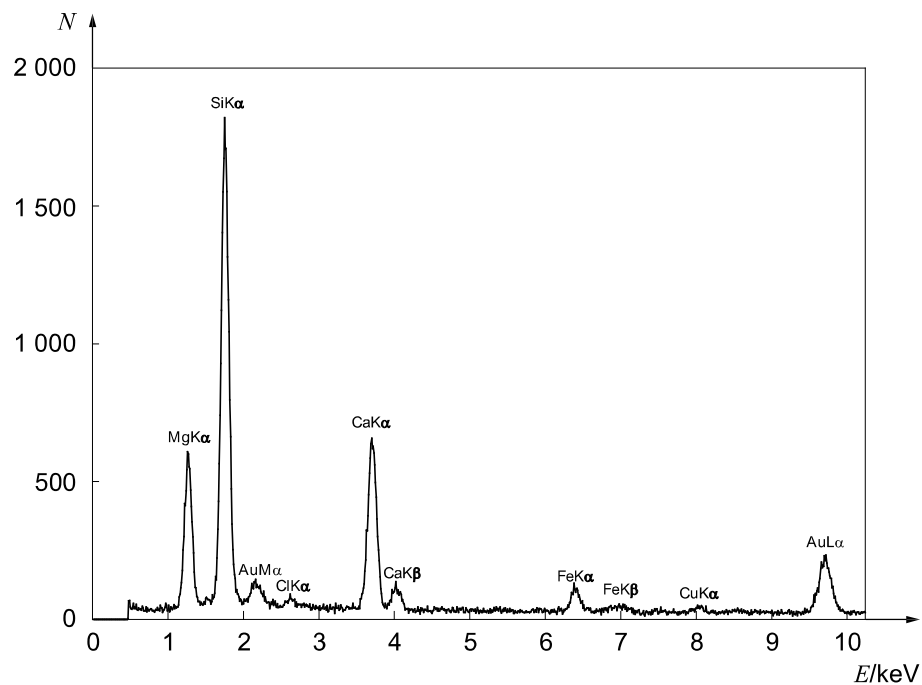
**Key**

N counts

E

X-ray energy

**Figure F.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite.
The gold peaks originate from the gold specimen grid**

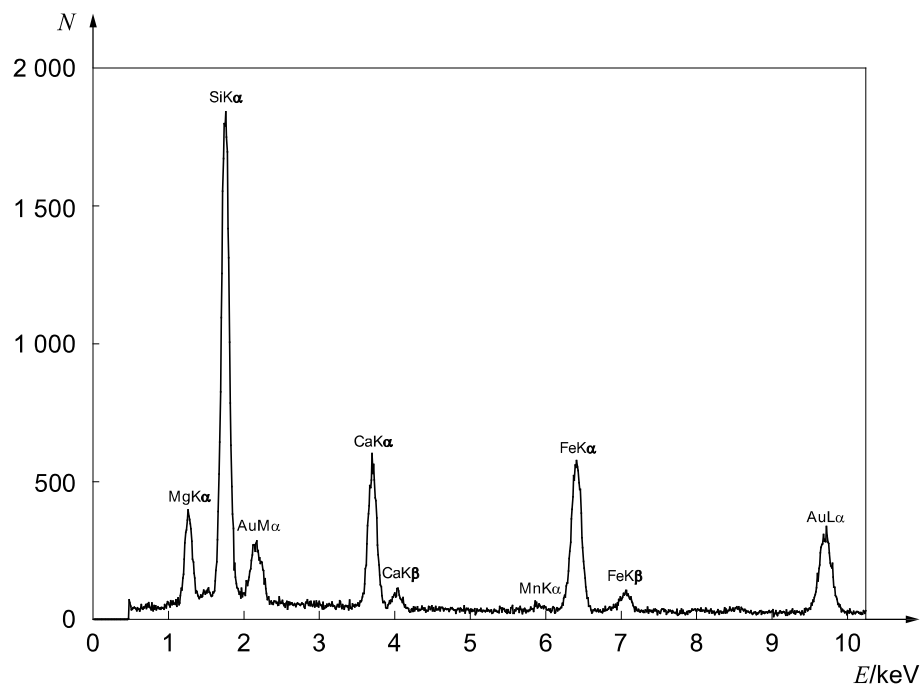
**Key**

N counts

E

X-ray energy

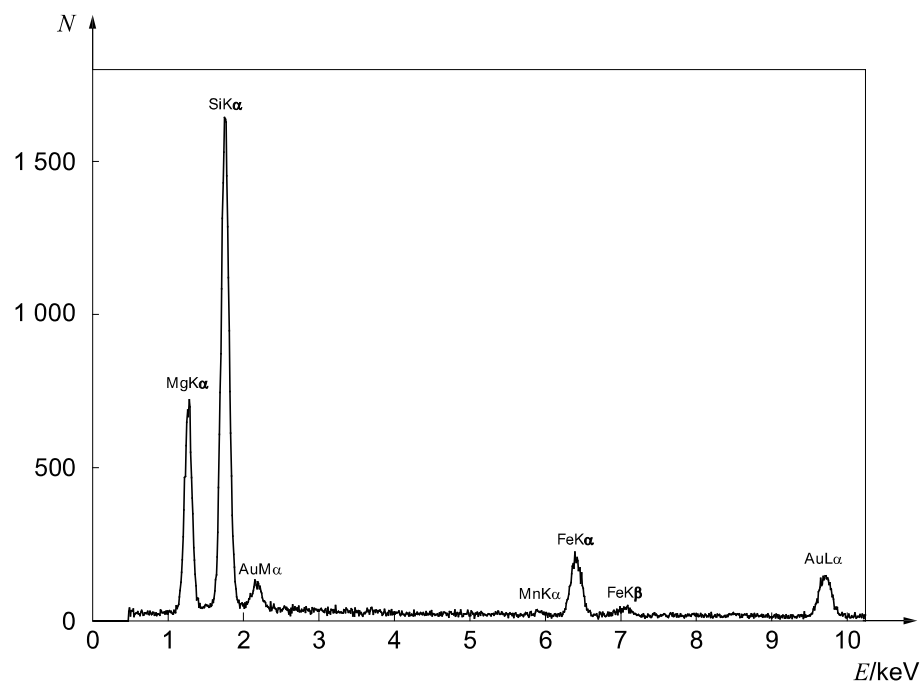
**Figure F.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite.
The gold and small copper peaks originate from the gold specimen grid**



Key

N counts E X-ray energy

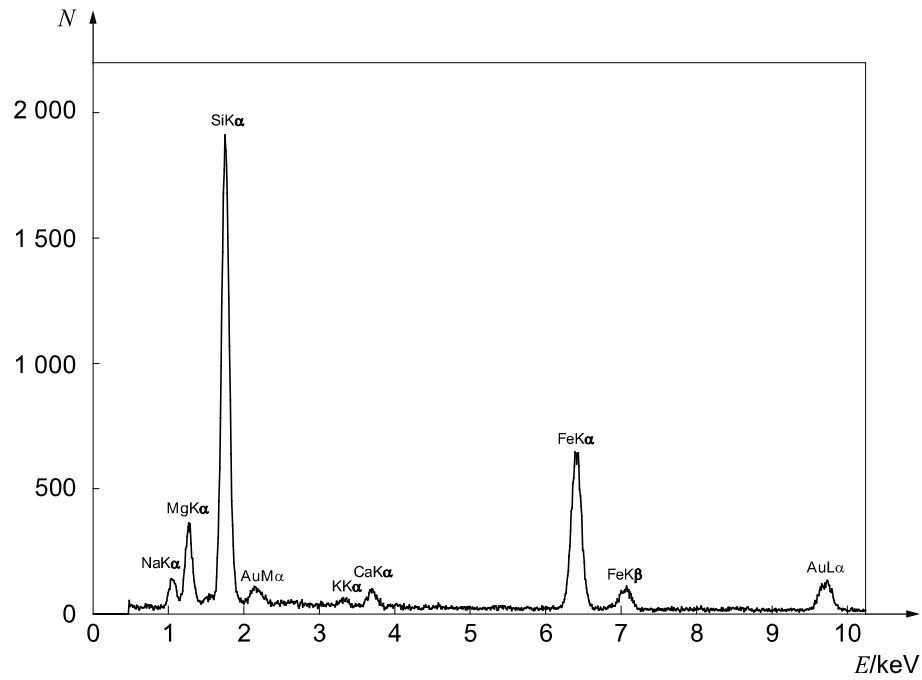
**Figure F.8 — Energy dispersive X-ray spectrum obtained from HSE actinolite.
 The gold peaks originate from the the gold specimen grid**



Key

N counts E X-ray energy

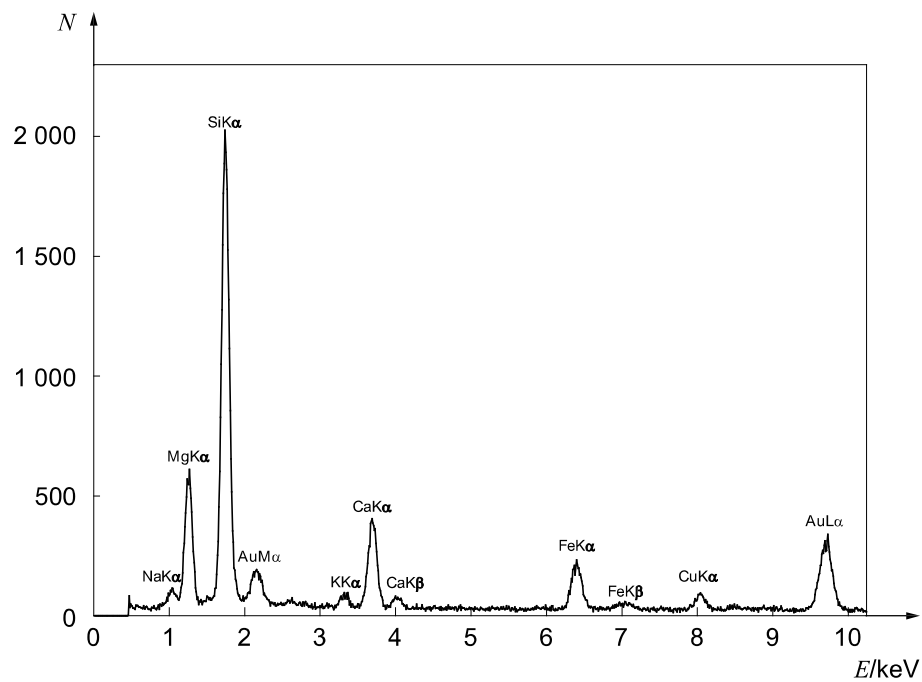
**Figure F.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite.
 The gold peaks originate from the gold specimen grid**

**Key**

N counts

E X-ray energy

**Figure F.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite.
The gold peaks originate from the gold specimen grid**

**Key**

N counts

E X-ray energy

**Figure F.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite asbestos.
The gold and small copper peaks originate from the gold specimen grid**

F.3 Electron diffraction

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel to the incident electron-beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a zone-axis ED pattern. In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but it is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone axis. The angle between the two zone axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focused in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it is necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, it is recommended that an internal calibration standard be used. Apply a thin coating of gold, or other suitable calibration material, to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information. Alternatively, a calibrated objective aperture can be inserted to determine if the layer-line spacing of the ED pattern is approximately 0,53 nm, as expected for asbestos fibres (Reference [30]). This works well even when viewing a raised screen through binoculars.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

ED patterns can be particularly useful for differentiating fibrous talc from anthophyllite asbestos, both of which have similar EDXA spectra. ED of talc produces a pseudo-hexagonal pattern that does not change as the fibre is tilted using the goniometer. Anthophyllite asbestos, on the other hand, produces assorted spots appearing and disappearing along layer lines as the fibre is tilted using the goniometer. ED patterns can also be a useful diagnostic tool for chrysotile that is so heavily coated with matrix that EDXA is inconclusive. Detection of the 002, 110, and 130 reflections as shown in Figure F.12 in conjunction with 0,53 nm layer-line spacing confirms the presence of chrysotile.

Analysis of laboratory samples seldom requires zone-axis measurements. However, if a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED pattern appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis

conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple

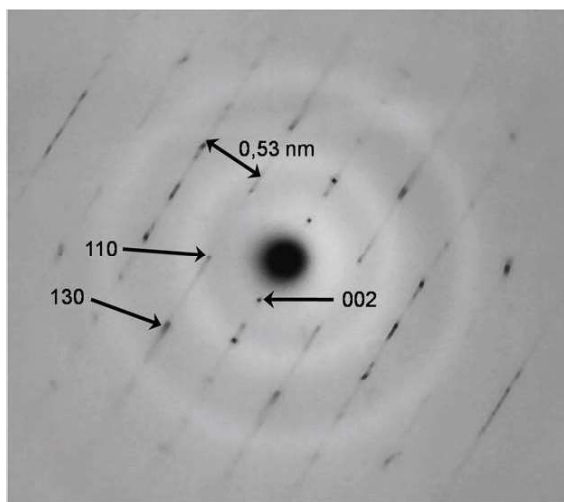


Figure F.12 — Chrysotile SAED pattern

diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in References [26]–[29].

It is important to recognize that not all zone-axis patterns that can be obtained are definitive. Only those patterns with closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all d -spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the smallest ring of the gold diffraction pattern (111), and that patterns with smaller distances between reflections are usually the most definitive. It is particularly important to recognize that when ED is used to discriminate between different minerals of similar compositions, demonstration that an ED pattern is consistent with the crystal structure of a particular mineral is not proof of identity, unless the ED pattern has also been shown to be *inconsistent* with the crystal structures of the other possible minerals.

Computer programs such as XIDENT (Reference [31]) provide a convenient way to test the consistency of any given ED pattern with the crystallographic data for individual minerals. The XIDENT program is advantageous in that no knowledge of crystal orientation is required; all possible ED patterns at all orientations are calculated and compared with the observed ED pattern. If the results obtained from one ED pattern do not resolve any ambiguity in identification of a fibre, a second ED pattern obtained at a different orientation of the fibre can be examined, and the observed tilt angle between the two orientations can be compared with the theoretical angle calculated from the suspected crystal structure. In order to use the XIDENT program, five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern are selected for measurement, as illustrated in Figure F.13. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very over-exposed, it does not provide a well-defined origin for these measurements. The required distances are best obtained by measuring between pairs of spots symmetrically disposed about the centre spot, preferably separated by several repeat distances.

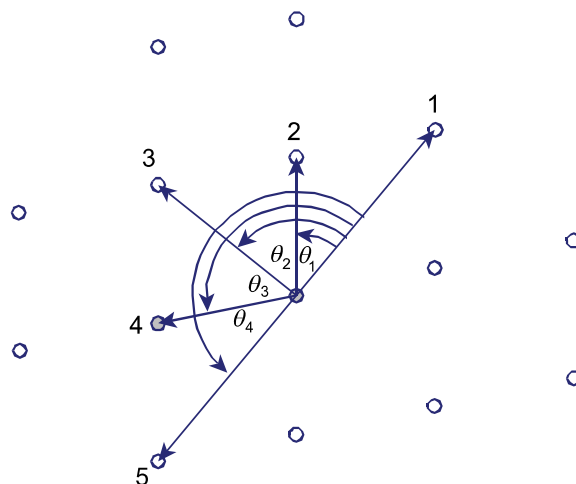


Figure F.13 — Measurement of spacings and angles in a zone axis ED pattern

Annex G
(informative)**Example of sampling record**

Date:	Samples taken by:
Building and location:	

Room:		Sample identification:
Sampling location:		
Reference:	Plan No:	Position in plan:
Sketch No:		Photo No:
Sample details:		
Comments:		

Annex H (informative)

Example of test report

Analysis of bulk materials for asbestos by ISO 22262-1

Date of analysis:			
Analyst:		Signature:	
<p>NOTE ISO 22262-1 refers to qualitative analysis of commercial products for asbestos.</p> <p>In this method, polarized light microscopy with dispersion staining is the default procedure for identification of asbestos. If the sample characteristics required the use of either of the optional electron microscope methods to identify asbestos, the method used is indicated. If accurate quantification of asbestos mass fraction in the range below approximately 5 % mass fraction is required for the purpose of determining the regulatory status of an asbestos-containing material, use the appropriate other parts of ISO 22262.</p>			

Sample	Asbestos	Estimated asbestos mass fraction	Non-asbestos fibres	Comments
Sample 20050411-1 Pipe covering Grey corrugated paper	Chrysotile	5 %–50 %	Cellulose Brucite	Sample ashed to remove interfering materials.
Sample 20050412-3 Pipe covering White fibrous material	Amosite Chrysotile	5 %–50 % 0,1 %–5 %	None	
Sample 20050412-4 Fireproofing from beam Blue fibrous material	Crocidolite	50 %–100 %	None	
Sample 20050413-1 Pipe covering Off-white fibrous material	None detected	0 %	Mineral wool	
Sample 20050413-2 Plaster White material	Tremolite	0,1 %–5 %	None	
Sample 20050413-3 Ceiling tile Grey fibrous material	Chrysotile	0,1 %–5 %	Mineral wool Cellulose	Chrysotile too fine to identify by PLM. Chrysotile identified by TEM method.

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